

# Adhesive Cementation of Etchable Ceramic Esthetic Restorations

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## ABSTRACT

This article describes the different materials and techniques that are used for adhesive cementation. Particular attention is given to treatments suitable for dentin, as well as the selection of surface treatments for various restorative materials. Factors related to the durability and stability of the adhesive process, as well as the clinical and laboratory procedures required for cementation, are also discussed.

For citation purposes, the electronic version is the definitive version of this article: [www.cda-adc.ca/jcda/vol-75/issue-5/379.html](http://www.cda-adc.ca/jcda/vol-75/issue-5/379.html)

A variety of restorative techniques with minimal invasion of the dental tissues have been reported. These techniques preserve tooth structure and reduce the possibility of damage to the pulp. As a consequence, the development of new restorative materials has been based on the concept of micro-retention (rather than macro-retention), which allows better conservation of the dental structure, provided appropriate adhesive procedures are used.<sup>1</sup>

The adhesive concepts that have been used for direct restorative procedures are now being applied to indirect restorations and have been incorporated into daily practice. The success of nonmetallic restorations depends primarily upon the luting agent, which must guarantee an effective, durable bond between the restoration and the dental structure; the luting agent is also responsible for marginal integrity.<sup>1</sup> Luting agents must have low solubility and low radiopacity, they must yield good esthetic results, and they must be biocompatible.<sup>2-4</sup>

Several studies<sup>1,3-5</sup> have investigated some of the relevant properties of resin luting ma-

terials, such as bond strength, degree of conversion and wear, to predict clinical behaviour. This article describes the materials and techniques used in adhesive cementation and the pre-cementation surface treatments required for different types of restorative material.

## Polymerization of Resin Luting Cement

Resin cements are divided into 3 groups according to polymerization process: chemically activated cements, which are used for Maryland bridges and intraradicular posts; light-cured cements, which are used for veneers; and dual-cured cements, which are used for inlays, onlays and crowns. Light-cured resin cements have the clinical advantages of longer working time and better colour stability.<sup>1</sup> Dual-cured resin cements have the advantages of controlled working time and adequate polymerization in areas that are inaccessible to light. They contain a peroxide-amine component for chemical polymerization and camphorquinone for light activation. They are usually composed of dimethacrylate monomers, such as bisphenol A glycidyl

methacrylate (BIS-GMA), urethane dimethacrylate (UDMA) or triethyleneglycol dimethacrylate (TEGDMA), and inorganic filler particles, which help to reduce the thermal expansion coefficient and polymerization shrinkage and thereby increase resistance to wear.<sup>6</sup>

The polymerization process for dual-cured resin cements has been investigated by several authors.<sup>7-9</sup> Specimens prepared with dual-cured cements and cured in the absence of light exhibited poorer mechanical properties than those that underwent light-curing.<sup>7,8,10</sup> Photoactivation increased the degree of conversion and surface hardness and reduced the wear of dual-cured cements.<sup>1,10,11</sup>

The use of resin luting agents has been encouraged for all-ceramic restorations because of their low solubility, good esthetics and high bond strength; however, longitudinal studies have shown marginal degradation over time due to wearing of the resin cement.<sup>4,12-16</sup> Cements with greater amounts of filler had less wear, a factor that may facilitate the clinician's choice of resin cement.<sup>1</sup>

### Surface Treatment for Etchable Ceramic Restorations

Successful adhesion depends on proper cleaning of the internal surfaces of the restoration to allow a strong link between the cement and the restoration. Some agents increase the surface energy on the restoration, thereby promoting an effective bond with the resin cement.

The strength and durability of the bond at the interface between ceramic and resin cement depends on the type of treatment selected, which is in turn governed by the microstructure of the ceramic material. Conventional ceramics have silica ( $\text{SiO}_2$ ) and potash feldspar ( $\text{K}_2\text{O}$ ,  $\text{Al}_2\text{O}_3$ ,  $6\text{SiO}_2$ ) or/and soda feldspar ( $\text{Na}_2\text{O}$ ,  $\text{Al}_2\text{O}_3$ ,  $6\text{SiO}_2$ ) as basic components. They are rich in the glass phase and have shown high bonding strength to resin cements.<sup>17-19</sup> Mechanical union is achieved by sandblasting with aluminum oxide particles and conditioning with hydrofluoric acid (HF). Silane ( $\text{SiH}_4$ ) promotes additional chemical bonding.<sup>18,19</sup> The use of hydrofluoric acid exposes the crystals at the surface of the ceramic structure, creating areas of microretention. The result of the chemical reaction between hydrofluoric acid and the silica phase of the feldspathic ceramics is a salt named hexafluorosilicate, which is removed by water spray.<sup>20</sup>

Infiltration of the resin cement into the micropores created by the acid is the key factor in the bonding between the ceramic and the luting agent. Furthermore, silane, which is responsible for the chemical union, is a bifunctional molecule, reacting with the inorganic particles of ceramics through the inorganic radical (OH group) and copolymerizing with the resin cement through the organofunctional radical (methacrylate group).<sup>4</sup> Effective silane action depends on hydrolysis by a weak acid. Single-bottle forms of silane are prehydrolyzed and have

a shorter shelf-life and hence reduced effectiveness over time. Double-bottle solutions are preferred.<sup>21</sup>

When surface treatments commonly used for feldspathic ceramics are applied to ceramics with high alumina ( $\text{Al}_2\text{O}_3$ ) content (e.g., Procera, Nobel Biocare, Göteborg, Sweden; In-Ceram, Vita Zahnfabrik, Bad Säckingen, Germany), the results are poor.<sup>22</sup> The scarcity of the glass phase permits neither exposure of crystals at the ceramic structure nor chemical reaction with the silane, which results in a weak bond with the resin cement.<sup>22</sup> Therefore, the composition of the particular ceramic system should be considered before the surface treatment is selected. Depending on the glass-phase content of the ceramic, it may be sensitive to acid-etching (as is the case for feldspathic ceramic, feldspathic ceramic reinforced with leucite and feldspathic ceramic reinforced with lithium disilicate) or resistant to acid (as for aluminum oxide ceramics, aluminum oxide ceramics reinforced with zirconium oxide and zirconium oxide ceramics)<sup>23</sup> (Table 1).

Sandblasting is an alternative conditioning procedure for aluminum oxide ceramics with minor or no glass content. Given the greater fracture resistance of these ceramics, they can be cemented to dentin using a glass ionomer or zinc phosphate cement.<sup>23</sup> Another alternative is to use a silicoating procedure, which promotes a durable bond with high-content alumina or zirconium ceramics.<sup>24,25</sup>

### Treatments for Dentin

Since adhesive systems were first introduced to operative dentistry, their formulations have undergone significant modifications to increase bond strength and simplify application. However, the great variety of adhesive agents now available has made it difficult for practitioners to choose the best system for a given patient. The following factors must be considered.

Application of phosphoric acid increases the surface energy of the dentin by removing the smear layer and promoting demineralization of the most superficial layer of hydroxyapatite crystals. The resin monomers infiltrate the water-filled spaces between collagen fibres, which results in a hybrid layer composed of collagen, resin, residual hydroxyapatite and traces of water, as first described by Nakabayashi and others in 1982.<sup>26</sup> Total etching is recommended as the first step for the 2- and 3-step adhesive systems.<sup>27</sup>

To reduce the number of operative steps and to simplify the clinical procedures, self-etching adhesive systems, which do not require a separate acid-etching step, have been introduced to the market. With these systems, an acidic primer partially dissolves the smear layer and hydroxyapatite crystals, thereby creating a hybrid layer that incorporates crystals and smear.<sup>27</sup> Self-etching systems can be classified in 2 categories: systems in which

**Table 1** Surface treatment protocols according to ceramic composition

Material	Brands	Procedure
Feldspar ceramics	Duceram, Degussa Dental GmbH, Hanau, Germany	1. Sandblast with 30- to 50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ particles (at 80 psi). 2. Etch with 9.5% hydrofluoric acid for 2–2.5 min, then wash and dry. 3. Apply silane for 1 min and dry.
Leucite-reinforced ceramic	IPS Empress, Ivoclar-Vivadent, Schaan, Liechtenstein	1. Sandblast with 30- to 50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ particles (at 80 psi). 2. Etch with 9.5% hydrofluoric acid for 60 s, then wash and dry. 3. Apply silane for 1 min and dry.
Lithium disilicate-reinforced ceramic	IPS Empress 2, Ivoclar-Vivadent	1. Sandblast with 30- to 50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ particles (at 80 psi). 2. Etch with 9.5% hydrofluoric acid for 20 s, then wash and dry. 3. Apply silane for 1 min and dry.
Glass-infiltrated aluminum oxide ceramic <sup>a</sup>	In-Ceram alumina, Vita, Bad Säckingen, Germany	1. Sandblast with synthetic diamond particles or 30- to 50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ particles (at 80 psi).
Zirconium-reinforced ceramic <sup>a</sup>	In-Ceram alumina, Vita	1. Sandblast with synthetic diamond particles or 30- to 50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ particles (at 80 psi).
Aluminum oxide ceramic <sup>a</sup>	Procera, Nobel Biocare, Göteborg, Sweden	1. Sandblast with synthetic diamond particles or 30- to 50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ particles (at 80 psi).

<sup>a</sup>To be used with resin cement, either phosphate-monomer-containing resin (first choice) or conventional resin cement (second choice). Alternatively, glass ionomer or zinc phosphate may be used.

application of the acidic primer is followed by application of a bonding resin (2 solutions) and systems with all-in-one (one-step) adhesives, which incorporate etchant, primer and bonding resin to a single solution.<sup>28</sup> Self-etching systems are considered less aggressive than phosphoric acid and may not bond as well to intact enamel and sclerotic dentin.<sup>29</sup>

Simplified adhesive systems are becoming more popular because of their simplicity and rapid application. However, the composition of these simplified versions is significantly modified, with greater quantities of acidic monomers, diluents and water. With these new formulations, the adhesive agents are more hydrophilic and therefore more susceptible to water sorption, which leads to hydrolytic degradation.<sup>28</sup>

Despite the increasing popularity of these simplified (i.e., one-bottle or all-in-one) adhesive systems, recent studies have demonstrated that dual-cured or chemically activated resin luting agents are subject to adverse chemical reactions and increased permeability when used in association with these systems. As a result, the bonding between the adhesive and the luting agent is weak.<sup>28</sup>

In particular, when simplified adhesive systems are used, the acidic groups from the outer layer of the adhesive that are not polymerized (because of the presence of oxygen) compete for peroxides with the tertiary amines of the resin luting agent. This results in an acid–base reaction between the adhesive system and the resin cement, which in turn prevents appropriate copolymerization.<sup>21</sup>

In addition, the hydrophilic characteristics of these simplified systems increase their susceptibility to the effects of water, leading them to behave as semipermeable membranes after polymerization.<sup>28,30</sup> The movement of water through the adhesive–dentin interface is facilitated by the presence of a hypertonic layer. The high concentration of calcium and phosphate ions in the hypertonic layer leads to a gradient of osmotic pressure, which causes movement of water from an area with high water content (the dentin tubules of the hydrated dentin) to an area with low water content (the adhesive–composite interface).<sup>30</sup> If the curing of dual-cured luting cements is delayed by the absence of light, there will be time for adverse chemical reactions to occur. In addition, the water that has undergone transudation from the dentin will accumulate at the interface, weakening bond strength in this area.<sup>30</sup>

These adverse effects can be minimized by selecting a conventional 3-step adhesive or 2-step self-etching system for use with dual-cured or chemical-cured luting cements. These adhesive systems have an additional non-acidic resin layer that is relatively hydrophobic; it can be applied during the second or third step of the system. This additional layer is impermeable and is chemically compatible with both dual-cured and chemical-cured resin cements; therefore, it will not promote any adverse reaction with tertiary amines from the luting agents and will reduce the permeability of the adhesive to water from the dentin.<sup>28</sup>

**Box 1** Clinical protocol for luting procedure with adhesive technique

1. Evaluate the prefabricated restoration on the stone cast for marginal adaptation and proximal contacts.
2. Remove the patient's provisional restoration.
3. Clean the cavity and try the inlay or onlay. First, check the proximal contacts of the restoration and then the marginal adaptation. Do not check the occlusion until you are finishing the cementation procedure.
4. Place a rubber dam.
5. Treat the internal surface of the inlay or onlay as appropriate for its composition (**Table 1**).
6. Apply bonding agent to the internal surface without curing, if this is specified by the manufacturer.
7. Etch the cavity with 35% or 37% phosphoric acid ( $H_3PO_4$ ) (15 s for dentin, 30 s for enamel). Gently wash and dry. Do not dehydrate the dentin.
8. Apply a thin layer of bonding agent on the cavity without curing.
9. Mix the base and catalyst of the resin cement, and apply to the restoration and the cavity.
10. Insert the restoration into the cavity and remove excess cement.
11. Apply light-curing for 10 s, and then remove excess resin cement from the proximal area (with dental floss).
12. Apply light-curing for 40–60 s per surface.
13. Remove rubber dam and check occlusion. Adjust if necessary.
14. Finish and polish with fine diamonds and rubber points.

**Cementation Technique**

The following clinical case of adhesive cementation of a crown illustrates the cementation protocol. The cementation technique is a relatively simple procedure, although it may seem complex because of the numerous clinical steps and the need to choose the best materials and technique for activating the surfaces before bonding. The cementation technique is outlined in **Box 1**, and **Table 1** lists the surface-treatment protocols for an etchable ceramic restoration.

After testing the adaptation of the restoration according to the protocol in **Box 1**, the ceramic restoration should be treated following the steps appropriate to the particular material, as listed in **Table 1**. The internal surface is etched with 9.5% hydrofluoric acid (**Figs. 1** and **2**) and then treated with silane (**Fig. 3**).

The tooth should be isolated (with either a rubber dam or a gingival retraction cord) to help control moisture from the gingival crevicular fluid (**Fig. 4**). Protecting the adjacent teeth is optional but can easily be achieved by applying Teflon tape (**Fig. 5**). The tooth is then etched with 37% phosphoric acid (15 s for dentin, 30 s for enamel) (**Fig. 6**) and rinsed. A bonding agent is then applied to the tooth and the resin cement is applied to the restoration (**Fig. 7**).

The colour match between the resin cement and esthetic restoration is usually excellent, which makes it difficult to remove all of the excess cement after curing is complete.<sup>31</sup> Therefore, an initial photoactivation period of 10 s at the occlusal surface is suggested, to stabilize the restoration in position. The gingival retraction cord can then be removed along with any excess cement (especially from the proximal areas), before the final photoactivation, with curing of at least 40 s for each surface<sup>9</sup> (**Fig. 8**).

Light-cured and dual-cured adhesive systems are available for indirect procedures. The use of an adhesive that must be light-cured before insertion of the indirect restoration is controversial, since the curing process may generate an adhesive layer that could interfere with adaptation of the restoration. In a recent study, Santos and others<sup>32</sup> evaluated the influence of some pre-cured adhesive systems on film thickness and the microtensile strength of bonding to dentin with indirect inlay restorations. Using scanning electron microscopy, these authors found that without pre-curing, none of the adhesive films could be distinguished from the resin luting layer. Pre-curing the adhesive produced a thicker adhesive film, with the thickness depending on the region along the internal border of the indirect restoration; however, pre-curing had no effect on bond strength for many of the adhesive systems tested.

Conventional ceramics are very fragile before cementation<sup>4</sup>; therefore, once the cementation procedure is complete, occlusion should be checked to ensure that the ceramic remains intact. The adhesive cementation technique provides additional reinforcement to both the restoration and the dental tissue because of the effective adhesion achieved at the cement–restoration and cement–dentin interfaces, which allows effective distribution of the occlusal forces along the restoration and dental structure.<sup>4</sup> This strengthening effect of the adhesive cementation technique on all-ceramic restorations has been reported in several studies.<sup>33–35</sup>

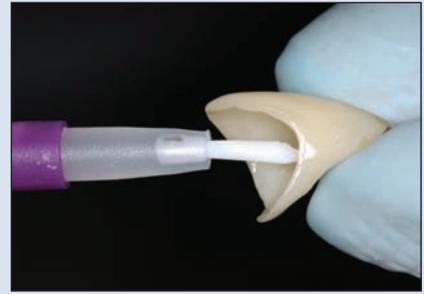
After occlusal adjustments, the surfaces should be carefully polished with diamond polishing gel applied



**Figure 1:** After being sandblasted with 50- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles, the internal surface of the crown is etched with 9.5% hydrofluoric acid for 20 s (see **Table 1**).



**Figure 2:** View of the internal surface of a crown after etching with 9.5% hydrofluoric acid. The crown is opaque white.



**Figure 3:** Silane is applied to the crown.



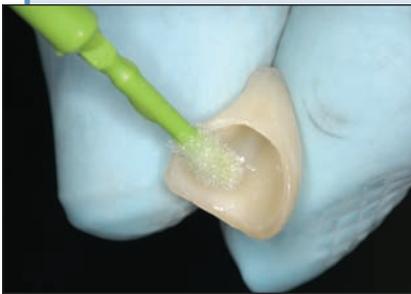
**Figure 4:** A gingival retraction cord is placed.



**Figure 5:** The adjacent teeth are protected with Teflon tape.



**Figure 6:** The tooth is etched with 37% phosphoric acid.



**Figure 7:** A resin cement is applied.



**Figure 8:** The gingival retraction cord is removed.



**Figure 10:** Final result.

with rubber tips and felt discs, to avoid any irregularities that might predispose the tooth to cracking<sup>36</sup> (**Fig. 9**). Follow-up appointments are indicated to evaluate these restorations. The application of a surface resin sealant at the supragingival borders of inlays and onlays can minimize wearing of the cement and reduce marginal degradation.<sup>37</sup>

### Conclusions

Adhesive cementation is a complex procedure that requires knowledge of adhesive principles and meticulous adherence to the clinical protocol to maximize

the bonding between tooth structure and restorative material. The application of an appropriately selected adhesive material with proper technique will ensure predictable results and successful long-term clinical outcomes. ✦

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The authors have no declared financial interests in any company manufacturing the types of products mentioned in this article.

This article has been peer reviewed.

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